

Kinetics for the Removal of Reactive Blue 2 from Aqueous Solution on the Nano Porous carbon materials Prepared from *Balsamodendron caudatum* Wood Waste

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Abstract

Balsamodendron caudatum wood waste was employed for the removal of Reactive Blue 2 from aqueous solution under different experimental conditions was investigated. Characterization of activated carbon prepared using *Balsamodendron caudatum* wood waste by two different processes (BAC1 and BAC2) was employed. The influences of initial Reactive Blue 2 concentration (20, 40 and 60 mg/L), and temperatures (30°, 45° and 60°C) have been reported. A comparison of kinetic models applied to adsorption of Reactive Blue 2 (CI No 61211) on the each adsorbent was evaluated for pseudo first-order, pseudo second-order and Elovich models respectively. Results show that the pseudo first order kinetic model was found to correlate the experimental data well.

Keywords: Adsorption Reactive Blue 2; Adsorption kinetics; low-cost adsorbents; Aqueous solution.

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1. INTRODUCTION

Reactive Blue 2 present in the effluents produced during the electroplating, paper industries, mining, dyeing, and photographic industries and causes severe environmental and public health problems. Reactive Blue 2 has been reported to be toxic to animals and humans and it is known to be carcinogenic. The concentration of Reactive Blue 2 in industrial wastewater is in the range 0.06 to 2.68 mg/L (Patterson 1985). The tolerance limit for Reactive Blue 2 for discharge into potable water is 0.050 mg/L in order to comply with this limit, it is essential that industries treat their effluents to reduce the Reactive Blue 2 to acceptable level. Many reports have appeared on the development of lowest activated carbon adsorbents developed from cheaper and readily available materials (Babel and Konawa 2003).

Activated nanoporous carbon with large surface area, micro porous character and chemical nature of their surface have made them potential adsorbents for removal of heavy metals from industrial wastewater. The adsorptive properties of active carbon for removal of pollutants are well documented (Macias et al. 1993). Adsorption of hazardous soluble chemicals from wastewater in to surface of a solid adsorbent has

provided a new dimension to wastewater technology (Benfield et al. 1982).

One of the major challenges associated with adsorption by activated carbon is its cost effectiveness. Hence research of recent past mainly focused on utilizing waste materials as alternatives to activated carbon. Rice husks (Youseff et al. 1990; Roy et al. 1993), fruit stones (Namasivayam and Periyasamy 1993; Lopez et al. 1984; Gharaibeh et al. 1998), coconut shells (Alaerts et al. 1989; Manju et al. 1998), fertilizer waste (Srivastava and Tyagi 1997, 1995), fly ash (Grover and Narayanaswamy 1982; Sell et al. 1994), peat moss (Allen and McKay 2001; Chen et al. 2001), ferronina shell (Karthikeyan et al. 2008) and ipomoea carnia stem waste (Karthikeyan et al. 2007) are some of the waste materials which have been fruitfully tried for this purpose. In this study, two activated nanoporous carbons prepared from *Balsamodendron caudatum* wood waste material (BAC1 and BAC2) are used to remove Reactive Blue 2 from aqueous solution using temperature, concentration and pH as parameters.

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2. EXPERIMENTAL

2.1 Adsorbent

Balsamodendron caudatum wood waste was obtained from various regions of Erode and Tirupur Districts, Tamil Nadu, India. The study of *Balsamodendron caudatum* wood waste material is used as adsorbent is expected to be economical, environmentally safe and it has practical importance. To develop adsorbents, the material was first ground and washed with doubly distilled water and then dried. The dried material thus obtained was treated with hydrogen peroxide (30% W/V) at room temperature for about 24 hrs to oxidize the adhering organic matter. The resulting material was thoroughly washed with doubly distilled water and then subjected to the temperature of 120°C for the moisture removal.

One portion of the above material was soaked well with H₂SO₄ solution for a period of 24 hours. At the end of 24 hrs the excess of H₂SO₄ solution were decanted off and air-dried. Then the materials were placed in the muffle furnace carbonized at 120-130°C. The dried materials were powdered and activated in a muffle furnace kept at 800°C for a period of 60 minutes. After activation, the carbon of obtained were washed sufficiently with large volume of water to remove free acid, Then the obtained material was washed with plenty of water to remove excess of acid, dried then to desired particle size. Another portion of the material was activated with activating agents Na₂SO₄ as per the H₂SO₄ activation process described above, and they sieved to desired particle size. Final products obtained in each case were stored separately in a vacuum desiccator until used. The resulting carbons named as (BAC1 and BAC2).

The N₂ adsorption-desorption isotherms of activated carbons were measured at 77K using a gas sorption analyzer (NOVA 1000, Quanta Chrome corporation) in order to determine the surface areas and the total pore volumes. The surface areas were calculated using the BET equation.

2.2 Batch adsorption studies

All reagents used were of AR grade (E merck). 50ml of Reactive Blue 2 solution of known concentration (C₀) and initial pH was taken in a 100ml screw-cap conical flask with a required amount of adsorbent and was agitated at a speed of 200 rpm in a thermostatic shaker bath at 27° C for a specified period of time. Then the solution was filtered through a 0.45 µm membrane filter.

2.3 Determination of Reactive Blue 2

Reactive Blue 2 was estimated spectrophotometrically. A calibration graph for 5-50mg of Reactive Blue 2 was prepared by the above procedure and concentration in the sample aliquot was established by referring to the calibration graph.

The amount of Reactive Blue 2 adsorbed in mg/L at time t was computed by using the following equation.

$$q_t = \frac{C_0 - C_t}{m_s} \times V \quad (1)$$

where, C₀ and C_t are the Reactive Blue 2 concentration in mg/L initially and at a given time t, respectively, V is the volume of the Reactive Blue 2 solutions in ml and m_s is the weight of the activated nanoporous carbon. The percentage of removed Reactive Blue 2 ions (R %) in solution was calculated using eqn. (2)

$$\% \text{ Removal} = \frac{C_0 - C_t}{C_0} \times 100 \quad (2)$$

The initial concentration of Reactive Blue 2, pH and temperature was investigated by varying any one parameters and keeping the other parameters constant.

2.4 Adsorption dynamics

The study of adsorption dynamics describes the solute uptake rate and evidently this rate controls the residence time of adsorbate uptake at the solid-solution interface. The kinetics of Reactive Blue 2 adsorption on the activated nanoporous carbons were analyzed using pseudo first order (Lagergren 1898), pseudo second order (Ho et al. 2000) kinetic models and Elovich equation (Chien and Clayton 1980). The conformity between experimental data and the model predicted values was expressed by the correlation coefficients (r² values closer or equal to 1). A relatively high r² value indicates that the model successfully describes the kinetics of Reactive Blue 2 adsorption.

2.5 The pseudo first – order equation

The pseudo first - order equation (Lagergren 1898) is generally expressed as follows.

$$\frac{dq_t}{dt} = k_1 (q_e - q_t) \quad (3)$$

where, q_e and q_t are the adsorption capacity at equilibrium and at time t , respectively (mg g^{-1}), k_1 is the rate constant of pseudo first-order adsorption (l min^{-1}).

After integration and applying boundary conditions $t=0$ to $t=t$ and $q_t=0$ to $q_t=q_e$, the integration form of equation (3) becomes.

$$\log(q_e - q_t) = \frac{\log(q_e) - k_1}{2.303} xt \quad (4)$$

The value of $\log(q_e - q_t)$ were linearly correlated with t . The plot of $\log(q_e - q_t)$ Vs t should give a linear relationship from which k_1 and q_e can be determined from the slope and intercept of the plot, respectively.

2.6 The pseudo second-order equation.

The pseudo second-order adsorption kinetic rate equation is expressed as (Ho et al. 2000)

$$\frac{dq_t}{dt} = k_2 (q_e - q_t)^2 \quad (5)$$

where, k_2 is the rate constant of pseudo second-order adsorption ($\text{g. mg}^{-1} \text{ min}^{-1}$). For the boundary conditions $t=0$ to $t=t$ and $q_t=0$ to $q_t=q_e$, the integrated form of equation (5) becomes.

$$\frac{1}{q_e - q_t} = \frac{1}{q_e} + k_1 \quad (6)$$

Which is the integrated rate law for pseudo second-order reaction. Equation (6) can be rearranged to obtain equation (7), which has a linear form.

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} (t) \quad (7)$$

If the initial adsorption rate h ($\text{mg g}^{-1} \text{ min}^{-1}$) is

$$h = k_2 q_e^2 \quad (8)$$

Then equations. (7) And (8) become:

$$\frac{t}{q_t} = \frac{1}{h} + \frac{1}{q_e} (t) \quad (9)$$

The plot of (t/q_t) and t of equation (7) should give a linear relationship from which q_e and k_2 can be determined from the slope and intercept of the plot, respectively.

2.7 The Elovich equation

The Elovich model equation is generally expressed (Chien and Clayton 1980) as

$$\frac{dq_t}{dt} = \alpha \exp(-\beta q_t)^2 \quad (10)$$

where, α is the initial adsorption rate ($\text{mg.g}^{-1} \text{ min}^{-1}$), β is the adsorption constant (g. mg^{-1}) during any one experiment.

To simplify the Elovich equation, assumed $\alpha\beta t \gg 1$ and by applying the boundary conditions $q_t=0$ at $t=0$ and $q_t=q_e$ at $t=t$ Eq (10) becomes;

$$q_t = \frac{1}{\beta} \ln(\alpha\beta) + \frac{1}{\beta} \ln t \quad (11)$$

If Reactive Blue 2 adsorption fits the Elovich model a plot of q_t vs $\ln t$ should yield a linear relationship with slope of $(1/\beta)$ and an intercept of $(1/\beta) \ln(\alpha\beta)$

3. RESULT AND DISCUSSION

3.1 Characterization of adsorbent

Physico-chemical characterizations of the adsorbents were presented in Table 1.

Table 1. Characteristics of the Activated Carbon BAC1& BAC2

Parameter	BAC1	BAC2
Bulk density (g/ml)	0.47	0.44
Ash content (%)	2.39	2.45
pH	6.4	8.21
Moisture content (%)	4.91	5.71
Surface area (m^2/g)	505	399.64
Solubility in water (%)	0.9	2.21
Solubility in 0.25M HCl (%)	1.19	1.52
Decolorizing power (mg/g)	40.3	22.5
Iodine number (mg/g)	453	248
pH _{zpc}	4.9	3.91
Carbonyl (mmol/g)	0.00	0.00
Lactonic (mmol/g)	0.038	0.032
Phenolic (mmol/g)	0.013	0.010
Acidic (mmol/g)	0.019	0.021
(mmol/g)	0.412	0.393

3.2 Optimum pH

The adsorption behavior of the dye on both the adsorbents was studied over a wide pH range of 2.0 -10.0. Fig.1 depicts that the pH significantly affects the extent of adsorption of dye over both adsorbents and a reduction the amount adsorbed with increasing pH was observed in both cases. Fig.1 also specifies that maximum uptake of the Reactive Blue 2 is observed at pH 6.0 on BAC1 as well as BAC2. The percentage of amount of dye adsorbed then increases up to pH 5.5 for BAC1 and pH 6.0 for BAC2. After this pH it remains almost constant in both cases. As the surface charge density decreases with an increase in the solution pH, the electrostatic repulsion between the positively charged metal and the surfaces of adsorbent is reduced, this causes more adsorption. More importantly higher pH can result in the higher adsorption of positively charged dye due to the surface complex formation of reactions and formation of colloids / precipitate with anions like carbonates, chlorides, hydroxides etc., (Krupadam *et al.*, 2003). When solution pH > carbon ZPC, surface of carbon is negatively charged and can attract dye from the solution. Apparently, the higher solution pH value, the more negative charges on carbon surface, the more attractive to positive charged dye on the nanoporous carbon surface.

3.3 Effect of concentration

The batch adsorption experiments were carried out by using three different concentrations of metal ion viz. 20mg/L, 40mg/L and 60mg/L at pH 5.5 at the reaction temperature of 30 °C were selected for each adsorbent. Fig. 2a and 2b clearly reveals the extend of adsorption of metal ion on both adsorbents increases linearly with increase in concentration of the adsorbent and then remains constant.

3.4 Effect of Temperature on kinetic rate constant and rate parameters

For both adsorption processes, adsorption experiments were now carried out with fixed initial dye concentration (20mg/L) at pH 5.5 and at different temperature viz. 30 °C, 45 °C and 60 °C. The analysis of the data in (Table 2) reveals that the influence of temperature of the Reactive Blue 2 has very little influence on the pseudo second order rate constants. The table 2 also reveals that the influence of the temperature of lead on Elovich and pseudo first order rate constant is neither appreciable nor little.

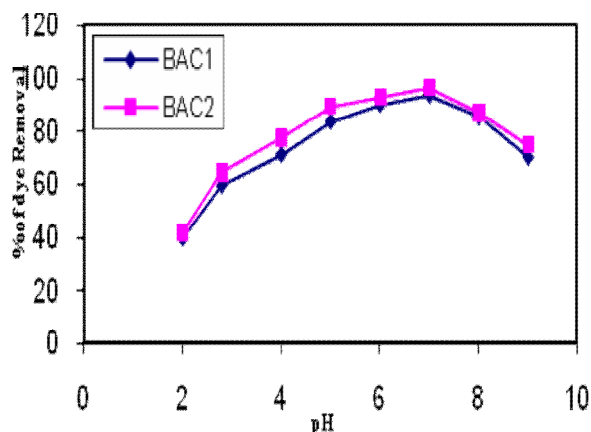


Fig. 1 : Effect of pH on percentage of removal of Reactive Blue 2 by BAC1 and BAC2

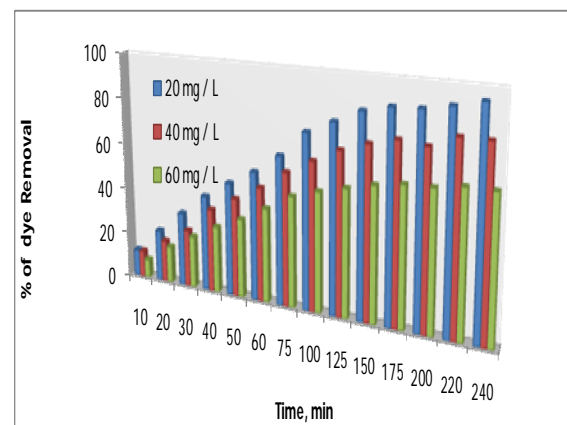
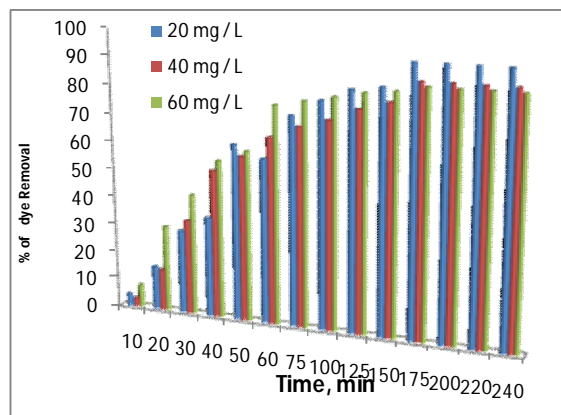


Fig. 2 : Effect of concentration on adsorption of Reactive Blue 2 over (a) BAC1 (b) BAC2 at different temperatures (pH5.5), adsorbent dosage.0.2g

Table 2. The adsorption kinetic model rate constants for BAC1 and BAC2 at different Temperature

Adsorbent	Initial Temperature, °C	Pseudo first order		Pseudo Second order			Elorich Model		
		k_1 l min^{-1}	r^2	k_2 $\text{g mg}^{-1} \text{min}^{-1}$	h $\text{mg g}^{-1} \text{min}^{-1}$	r^2	β g min^{-1}	α $\text{mg g}^{-1} \text{min}^{-1}$	r^2
BAC1	30	0.0175	0.9587	0.0055	0.3777	0.9833	0.1906	0.8594	0.9610
	45	0.0098	0.9576	0.0376	5.3055	0.9019	0.1734	0.4287	0.9211
	60	0.0254	0.9066	0.0087	0.1578	0.9156	0.1793	0.4637	0.9223
BAC2	30	0.4461	0.9774	0.0587	28.678	0.988	27.918	0.222	0.988
	45	0.1255	0.9780	0.0367	16.578	0.969	17.132	0.299	0.994
	60	0.3354	0.9365	0.2278	12.951	0.997	22.664	0.467	0.977

It is obvious that the adsorption of Reactive Blue 2 on the *Balsamodendron caudatum* wood waste activated carbon is best described by first order rate equation with regression coefficient value is greater than 0.98.

3.5 Thermodynamic Parameter

The thermodynamic parameters obtained for the adsorption systems were calculated using the following

$$\text{equations. } K_C = \frac{C_{Ae}}{C_e} \quad (12)$$

$$\Delta G = -RT \ln K_C \quad (13)$$

$$\log K_C = \frac{\Delta S}{2.303 R} - \frac{\Delta H}{2.303 RT} \quad (14)$$

K_C is equilibrium constant, C_{Ae} is the solid phase concentration at equilibrium, C_e is residual concentration at equilibrium, R is gas constant (J/mole) and T is the temperature in Kelvin

ΔH and ΔS was obtained from the slope and intercept of Vant Hoff plot ($1/T$ Vs $\ln K_C$). Table 3 gives the value of ΔG , ΔS and ΔH for the adsorption of BAC1 and BAC2. The negative values of free energy change (ΔG) indicate the feasibility and spontaneous nature of adsorption of BAC1 & 2. The positive value of ΔS is due to the increased randomness during the adsorption of adsorbents.

3.6 Mechanism

The high correlation coefficients obtained using pseudo first order and pseudo second order Models, it was impossible to concludes which adsorption mechanism actually occurred and was responsible for the ability of adsorbent to review other sources of information in an attempt to identify the specific adsorption mechanism.

In adsorption process of dye on the solid surface, the metal species migrate towards the surface of the adsorbent. This type of migration proceeds till the concentration of the adsorbate species, adsorbed, on to the surface of the adsorbent. Once equilibrium is attained, the migration of the solute species from the solution stops. Under this situation, it is possible to measure the magnitude of the distribution of the solute species between the liquid and solid phases. The magnitude of this kind of distribution is a measure of the efficiency of the chosen adsorbent in the adsorbate species.

When a powdered solid adsorbent material is made in contact with a solution containing dye, the dyes first migrate from the bulk solution to the surface of the liquid film. This surface exerts a diffusion barrier. This barrier may be very significant or less significant. The involvement of a significant quantum of diffusion barrier indicates the dominant role taken up by the film diffusion in the adsorption process. Furthermore, the rate of an adsorption process is controlled either by external diffusion, internal diffusion or by both types of diffusions.

Table 3. Thermodynamic parameters for Reactive Blue 2, BAC1 and BAC2 adsorption.

Adsorbent	$\Delta G \times 10^4 \text{ (KJ mol}^{-1}\text{)}$			ΔH	ΔS
	30°C	45°C	60°C	KJ mol ⁻¹	KJ mol ⁻¹
BAC1	-519.78	-900.98	-332.38	-21.6400	-56.113
BAC2	-11.77	-11.65	-9.50	13.15	211.65

The external diffusion controls the migration of the solute species from the solution to the boundary layer of the liquid phase. However, the internal diffusion controls the transfer of the solute species from the external surface of the adsorbent to the internal surface of the pores of the

adsorbent material (Gupta et al. 2005). It is now well established, that during the adsorption of metal ion over a porous adsorbent, the following three consecutive steps are taken place (Gupta et al. 2003).

- (i) transport of the ingoing adsorbate ions to external surface of the adsorbent (film diffusion).
- (ii) transport of the adsorbate ions within the pores of the adsorbent except for a small amount of adsorption, which occurs on the external surface (particle diffusion).
- (iii) adsorption of the ingoing adsorbate ions on the interior surface of the adsorbent.

Out of these three processes, the third process is considered to be very fast and is not the rate limiting step in the uptake of organic compounds (Weber Jr. et al. 1963). The remaining two steps impart the following three possibilities:

- Case 1: External transport > internal transport, where rate is governed by particle diffusion.
- Case 2: External transport < internal transport, where the rate is governed by film diffusion.
- Case 3: External transport \approx internal transport, which accounts for the transport of the adsorbate ions to the boundary and may not be possible within a significant rate, which later on gives rise to the formation of a liquid film surrounded by the adsorbent particles with a proper concentration gradient.

In the batch-mode contact time adsorption experiments, rapid stirring is maintained. This Reactive Blue 2 to the transport of the adsorbed species from the solution to the pores of the adsorbent material and this step may control the rate of the adsorption process.

pore diffusion coefficient, D_p , should be in the range 10^{-11} - 10^{-13} cm²/ sec. In order to find out the nature of the process responsible for adsorption on to chosen adsorbent, attempts were made to calculate the diffusion co-efficient of the process.

Assuming spherical geometry of the sorbents (Bhattacharya et al. 1984), the overall rate constant of the process can be correlated to the pore diffusion coefficient in accordance with the expression,

$$t_{1/2} = 0.03 \frac{r_o^2}{D_p} \quad (15)$$

or to the film diffusion coefficient in accordance with

$$t_{1/2} = 0.23 \frac{r_o \bar{C}}{D_f} \times \frac{\bar{C}}{C} \quad (16)$$

where r_o is radius of the sorbent (cm), D_p and D_f are pore diffusion coefficient (cm²/sec) and film diffusion coefficient (cm²/sec) respectively, $\frac{\bar{C}}{C}$ is equilibrium loading of the adsorbent, \bar{C} is the film thickness (cm) and $t_{1/2}$ is the time for half change (sec).

Since the carbon particles used were of the size range (180 - 250 μ m), the average diameter of the particle was taken as 0.0215×10^{-4} cm. Using these values, the film diffusion coefficients and pore diffusion coefficients were calculated. Then considering the pseudo first order rate constant k_L , for the adsorption of Reactive Blue 2. The values of D_p and D_f were calculated under the given set of operating conditions, and are presented in the Table 4.

Table 4. D_p and D_f values for the chosen adsorbent - adsorbate system

Temp, °C	$t_{1/2}$, s	BAC1		BAC2	
		$D_p \times 10^{-10}$ cm ²	$D_f \times 10^{-9}$ cm ²	$D_p \times 10^{-10}$ cm ²	$D_f \times 10^{-9}$ cm ²
30	4365.72	1.1510	4.722	1.454	5.265
45	4577.98	0.6892	3.635	0.965	3.977
60	4455.38	0.66341	3.143	0.676	2.376

According to Michelson et al., (1975) for film diffusion to be rate-determining step, the value of the film diffusion coefficient, D_f , should be in the range 10^{-6} - 10^{-8} cm²/ sec. If pore diffusion were to be the rate limiting, the The present study indicates the D_p values in the order of 10^{-11} to 10^{-10} cm²/sec and the D_f values in the order of 10^{-10} to 10^{-9} for the respective Lagergren plots for Reactive Blue 2 and hence, the investigator concludes that the mechanism of the removal of Reactive Blue 2 in the present study by both adsorbents is complex.

Since both external Mass transfer and intraparticle diffusion constants varied with initial Reactive Blue 2 concentration indicating the occurrence of both film diffusion and intra particle diffusion, the sorption data were further analyzed by the kinetic expression given by Boyd et al. (1953) as

$$F = 1 - \frac{6}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{n^2} \exp \left[\frac{-D t \pi^2 n^2}{r^2} \right] \quad (17)$$

$$F = 1 - \frac{6}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{n^2} \exp \left[-n^2 \beta_t \right] \quad (18)$$

Where F is the fractional attainment of equilibrium at time t and is obtained by using following equation and n. is the integer value.

$$F = \frac{q_t}{q_e} \quad (19)$$

Where q_t and q_e are the amounts adsorbed at time t and at equilibrium respectively.

On the basis of F values, corresponding values of B_t were obtained from equation 19 (Reichenberg, 1953).

The B_t vs time plots for the sorption of Reactive Blue 2 over BAC1 and BAC2 (Fig. 3a and 3b) are found to be linear in beginning. Thus the process seems to be particle diffusion controlled in this region, while during the later deviation from linearity occurs and they also did not pass through the origin through out concentration range at 30, 45 and 60°C. Thus the overall process can be interpreted as film diffusion. (D_i) were calculated at different temperatures using the following Equation.

$$B = \pi^2 D_i / r^2$$

From the slop of the straight line obtained from time versus B_t graph, the B value (time constant) was calculated. The values of effective diffusion coefficient

Where 'r' is the radius of the absorbent particle. The D_i values are given in the Table 5.

The plot of $1/T$ versus $\log D_i$ was found linear (not given) with negative slope indicating thereby the increase in the mobility of ions.

This is due to the fact that which consequently decrease the retarding force acting on with the rise in temperature the mobility of ions increases, the diffusing ions.

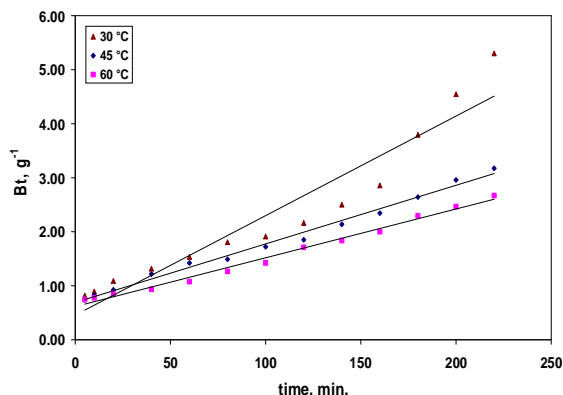


Fig.3a

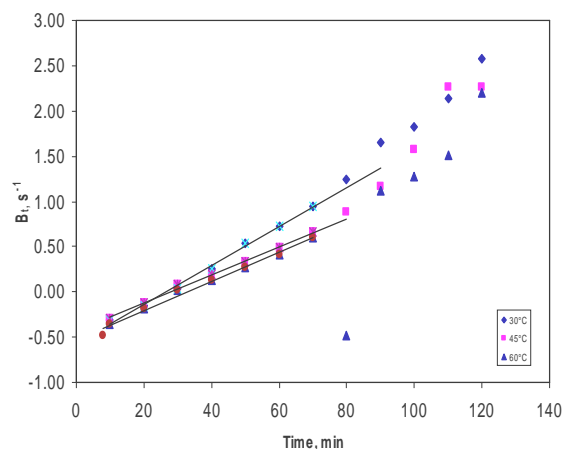


Fig.3b

Fig. 3 : Time vs B_t plots different temperature of (a) Reactive Blue 2 BAC1 adsorption and (b) Reactive Blue 2-BAC2 adsorption.

Table 5. Effective diffusion coefficient (D_i) pre exponential constants (D_0) activation energy (E_a) and entropy of activation ΔS^\ddagger for diffusion of Reactive Blue 2 in BAC1 and BAC2.

Adsorbent	$D_i (m^2/s)$			$D_0 (m^2/s)$	E_a KJ/mol	ΔS^\ddagger JK ⁻¹ mol ⁻¹
	30°C	45°C	60°C			
TAC1	2.12×10^{-13}	1.55×10^{-13}	4.25×10^{-13}	3.74×10^{-6}	40-8	-22.83
TAC2	3.5×10^{-13}	6.15×10^{-13}	8.66×10^{-14}	5.55×10^{-4}	26.4	-57.04

$$D_i = D_o \cdot \exp [-E_a / RT]$$

$$D_o = (2.72 d^2 kT/h) \cdot \exp [\Delta S^\# / R]$$

Where d is the average distance between the successive exchange sites and is taken as 5 Å. R, h and k are the Gas, Planck, and Boltzmann constants, respectively.

The values of E_a , D_i , D_o , $\Delta S^\#$ and other parameters are given in Table 4. The negative value of $\Delta S^\#$ reflects that no significant change occurs in the internal structure of chosen adsorbent during the adsorption process.

4. CONCLUSIONS

Removal of Reactive Blue 2 from aqueous solution was possible using several abundantly available low cost adsorbents. The adsorption of Reactive Blue 2 was found to be dependent on pH, temperature and concentration for both adsorbents. Thermodynamic parameters obtained for both the adsorbents accounts for feasibility of the process at each concentration. Further the kinetic studies apparently reveal that the removal takes through a film diffusion process at all the concentrations and temperature for BAC1 as well as BAC2. The percentage saturation was found to be almost 98.5 and 95.4 % for the BAC1 and BAC2 respectively. The kinetics of Reactive Blue 2 adsorption on different adsorbents was found to follow a pseudo first-order rate equation.

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